

## Determination of Total Particulate Matter, Nicotine, and Water in Cigarette Smoke\*

A collaborative study on cigarette smoke analysis was conducted during the year to test methods for collecting the smoke sample and to determine total particulate matter (TPM), total alkaloids reported as nicotine, and water in the smoke. The Analytical Methods Committee of the Tobacco Chemists Conference selected the methods to be tested. The directions included: a method for conditioning and selecting the samples to be smoked; the smoking machine characteristics, puff volume ( $40 \pm 0.5$  ml), duration ( $2 \pm 0.2$  seconds), and frequency ( $1/60 \pm 1$  second); traps and filter media

for collecting the smoke; and the analytical methods for determining the three materials, TPM, nicotine, and water.

Five cartons each of four different cigarettes made especially for this study were sent to each collaborator with a copy of the directions for the study. Each collaborator was provided with a report sheet and was asked to provide the following information:

- (1) Average weight of each type of cigarette.
- (2) Total number of puffs for each sample (10 cigarettes).
- (3) Weight of total particulate matter (wet) from each sample.
- (4) Weight of TPM (wet) per cigarette.
- (5) Weight of TPM (wet) per 100 ml of smoke.
- (6) Weight of "nicotine" per cigarette.
- (7) Weight of "nicotine" per 100 ml of smoke.
- (8) Weight of water per cigarette.

\* Presented by Referee C. L. Ogg at the Seventy-fifth Annual Meeting of the Association of Official Agricultural Chemists, Oct. 30–Nov. 1, 1961, at Washington, D.C.

<sup>1</sup> Liggett and Myers Tobacco Company, Durham, N.C.

<sup>2</sup> The American Tobacco Company, Richmond 24, Va.

<sup>3</sup> Philip Morris, Inc., Richmond 6, Va.

<sup>4</sup> Consolidated Cigar Corporation, Glastonbury, Conn.

(9) Weight of water per 100 ml of smoke.  
 (10) Weight of TPM (dry) per cigarette (weight of TPM/cigarette — weight of water/cigarette).

(11) Weight of TPM (dry) per 100 ml of smoke (weight of TPM/100 ml of smoke — weight of water/100 ml of smoke).

Collaborators were asked to furnish all data for each sample from each type of cigarette. The cigarettes were: Type A-70 mm unfiltered; Type B-85 mm unfiltered; Type C-85 mm filtered, low pressure drop; and Type D-85 mm filtered, high pressure drop. All cigarettes had the same tobacco blend and paper.

The cigarettes to be smoked were selected on a weight basis so that they were all within  $\pm 20$  mg of the average weight. They were conditioned for 2 days at  $75 \pm 2^\circ\text{F}$  and  $60 \pm 2\%$  relative humidity before selection for smoking. Two types of traps were used: a Cambridge CM113 fiber glass filter and an  $\alpha$ -cellulose filter, the latter being partially immersed in a Dry Ice-acetone bath. Ten cigarettes constituted a sample, and four samples of Type A and two of each of the other three types were smoked for each of the two filter systems. Because the same sample could not be used for both nicotine and water when the Cambridge filter was used, a total of 30 samples or 300 cigarettes were smoked by each laboratory. To obtain the necessary data, each laboratory had to conduct 60 analyses, 20 each for TPM, nicotine, and water.

### Results

The first measurement requested was determination of the average weight of each type of cigarette after conditioning at  $75 \pm 2^\circ\text{F}$  and  $60 \pm 2\%$  relative humidity. Table 1 shows the average weights obtained by each collaborator, the mean for 11 laboratories, and the interlaboratory standard deviation and coefficient of variation in per cent.

The interlaboratory reproducibility of the other measurements is shown in Table 2. Here the mean, the standard deviation, and coefficient of variation in per cent are calculated from the collaborator's mean values. The average coefficient of variation for the

four types of cigarettes for each smoke-trapping system is also shown.

The coefficients of variation for the number of puffs per sample, weight of TPM (wet) per cigarette, and nicotine per cigarette are all quite similar except for nicotine by the  $\alpha$ -cellulose trap, which is noticeably higher. These values are all considerably higher than had been hoped for, and they indicate the need for more work. One could not expect to obtain better precision in the TPM or nicotine determinations than is obtained in the sample collection. The fact

**Table 1. Average weight of conditioned cigarettes**

Coll. No.	Type A	Type B	Type C	Type D
1	0.954	1.177	1.178	1.097
2	0.978	1.193	1.182	1.080
3	0.984	1.186	1.192	1.104
5	0.988	1.173	1.181	1.088
8	0.988	1.226	1.199	1.124
9	0.988	1.187	1.159	1.082
11	0.999	1.204	1.199	1.121
20	0.963	1.196	1.172	1.081
22	0.977	1.18	1.18	1.09
23	0.994	1.221	1.163	1.095
24	0.991	1.199	1.191	1.106
Mean	0.982	1.195	1.181	1.097
Std Dev.	0.014	0.017	0.013	0.015
Coeff. of Var., %	1.4	1.4	1.1	1.4

that the precisions are all similar indicates that the TPM and nicotine methods in themselves are probably satisfactory.

When the results for TPM and nicotine were calculated on a smoke volume basis of 100 ml, the interlaboratory precisions were not as good as on the per cigarette basis. Thus rather than the variation in the number of puffs having been corrected, more variability was introduced; this result indicated a puff volume variability. Correlations between the number of puffs and the TPM and nicotine values have not been calculated, but inspection shows that these would be low.

The determination of water in the Cambridge filters was made so that the TPM

**Table 2. Reproducibility of results between laboratories**

	Cambridge Filter				$\alpha$ -Cellulose Filter			
	Type A	Type B	Type C	Type D	Type A	Type B	Type C	Type D
Number of Puffs per Sample								
Mean	79.4	105.1	108.9	102.3	81.8	107.5	109.8	106.3
Std Dev.	5.1	6.5	5.0	4.5	5.0	6.5	5.7	8.6
Coeff. of Var., %	6.4	6.2	7.3	4.4	6.1	6.0	5.2	8.1
Av. Coeff. of Var.		6.1				6.4		
Weight (mg) TPM (Wet) per Cigarette								
Mean	32.5	45.0	25.8	24.7	41.1	57.0	36.7	34.2
Std Dev.	1.70	1.94	1.76	1.90	1.88	2.56	1.88	2.61
Coeff. of Var., %	5.2	4.3	6.8	7.7	4.6	4.5	5.1	7.6
Av. Coeff. of Var.		6.0				5.5		
Weight (mg) TPM (Wet) per 100 ml of Smoke								
Mean	10.3	10.7	5.87	5.97	12.7	13.2	8.42	8.13
Std Dev.	0.84	0.65	0.40	0.52	1.12	0.90	0.82	1.09
Coeff. of Var., %	8.1	6.1	6.8	8.7	8.8	6.8	9.7	13.4
Av. Coeff. of Var.		7.4				9.7		
Weight (mg) Nicotine per Cigarette								
Mean	1.70	2.26	1.36	1.35	1.79	2.41	1.38	1.33
Std Dev.	0.13	0.15	0.08	0.09	0.26	0.25	0.16	0.14
Coeff. of Var., %	7.6	6.6	5.9	6.7	14.5	10.4	11.6	10.5
Av. Coeff. of Var.		6.7				11.8		
Weight (mg) Nicotine per 100 ml of Smoke								
Mean	0.54	0.53	0.31	0.33	0.55	0.56	0.32	0.32
Std Dev.	0.054	0.051	0.018	0.028	0.080	0.062	0.033	0.049
Coeff. of Var., %	10.0	9.6	5.8	8.5	14.5	11.1	10.3	15.3
Av. Coeff. of Var.		8.5				12.8		
Weight (mg) Water per Cigarette								
Mean	3.64	4.77	2.83	2.88	7.48	9.49	7.09	6.69
Std Dev.	1.16	1.46	1.06	1.22	2.80	1.64	2.16	2.31
Coeff. of Var., %	31.9	30.6	37.5	42.4	37.4	17.3	30.5	34.5
Av. Coeff. of Var.		35.6				29.9		
Weight (mg) TPM (Dry) per Cigarette								
Mean	28.8	40.1	22.9	21.8	34.7	46.5	29.6	27.4
Std Dev.	2.23	1.98	1.36	1.46	3.01	3.70	3.40	1.97
Coeff. of Var., %	7.7	4.9	5.9	6.7	8.7	8.0	11.5	7.2
Av. Coeff. of Var.		6.3				8.9		

**Table 3. Reproducibility of number of puffs per sample within laboratories**

Coll. No.	Cambridge Filter			$\alpha$ -Cellulose Filter		
	Type A		All <sup>b</sup>	Type A		All
	Std Dev.	C. V. % <sup>a</sup>	Std Dev.	Std Dev.	C. V. %	Std Dev.
1	1.90	2.38	2.95	1.55	1.85	1.41
2	1.29	1.63	1.66	2.93	3.57	3.84
3	2.75	3.53	1.48	1.80	2.25	1.55
5	3.30	4.40	2.61	1.30	1.67	1.90
9	0.81	0.96	1.55	3.87	4.45	3.01
11	1.55	1.85	1.55	1.16	1.45	0.45
20	1.00	1.39	1.76	0.81	1.04	1.34
22	1.52	2.00	1.70	1.00	1.28	1.38
23	0.58	0.64	0.84	0.58	0.62	0.95
24	0.96	1.26	1.00	1.50	1.95	1.64
Mean	1.57	2.00	1.56	1.65	2.01	1.59

<sup>a</sup> C. V. % = Coefficient of variation in %.<sup>b</sup> All = Standard deviation calculated from the differences between duplicates for all four samples.**Table 4. Reproducibility of TPM measurement within laboratories in mg per cigarette**

Coll. No.	Cambridge Filter			$\alpha$ -Cellulose Filter		
	Type A		All <sup>b</sup>	Type A		All
	Std Dev.	C. V. % <sup>a</sup>	Std Dev.	Std Dev.	C. V. %	Std Dev.
1	0.99	2.93	0.95	0.72	1.83	0.41
2	2.23	7.38	1.22	0.82	2.12	3.27
3	1.14	3.52	1.23	1.75	4.25	1.48
5	0.63	2.01	0.41	1.34	3.15	1.03
9	0.63	2.03	1.18	1.71	3.98	1.76
11	0.72	2.16	0.65	0.16	0.40	1.20
20	0.31	0.99	0.44	0.82	1.84	0.74
22	0.57	1.68	1.33	1.30	3.11	1.29
23	1.68	5.19	1.24	1.50	3.83	1.08
24	0.93	2.60	0.42	1.05	2.59	0.72
Mean	0.98	3.05	0.91	1.12	2.71	1.30

<sup>a</sup> C. V. % = Coefficient of variation in %.<sup>b</sup> All = Standard deviation calculated from the differences between duplicates for all four samples.

could be corrected for water. As the Cambridge filter does not retain all the water in the smoke, the moisture value does not reflect the water formed from the cigarette. The water values shown under  $\alpha$ -cellulose filter should be a measure of the water in the smoke, because all water vapor should be removed by the cold trap. The variability between laboratories for both filter systems was very high and more work will have to be done on this method. However, the

coefficient of variation for the TPM data corrected to the water-free basis was only a little higher than for the TPM on the wet basis.

The results of these tests for the inter-laboratory precision of the various measurements indicate a need for further work, particularly in standardization of the procedure or apparatus for collection of the smoke sample.

Within-laboratory precisions for the num-

**Table 5. Reproducibility of nicotine determination within laboratories in mg per cigarette**

Coll. No.	Cambridge Filter			$\alpha$ -Cellulose Filter		
	Type A		All <sup>b</sup>	Type A		All
	Std Dev.	C. V. % <sup>a</sup>	Std Dev.	Std Dev.	C. V. %	Std Dev.
1	0.21	11.17	0.13	0.48	20.25	0.36
2	—	—	—	0.038	2.64	0.066
3	0.020	1.11	0.046	0.024	1.43	0.046
5	0.056	3.64	0.027	0.014	0.86	0.012
9	0.11	7.48	0.082	0.080	4.68	0.13
11	0.018	1.07	0.019	0.026	1.54	0.021
20	0.029	1.77	0.039	0.049	2.93	0.047
22	0.13	7.22	0.19	0.028	1.49	0.042
23	0.040	2.29	0.037	0.082	4.63	0.044
24	0.048	2.65	0.023	0.068	3.37	0.057
Mean	0.073	4.27	0.047	0.088	4.38	0.083
c	0.050	3.40	0.037	0.040	2.61	0.047

<sup>a</sup> C. V. % = Coefficient of variation in %.<sup>b</sup> All = Standard deviation calculated from the differences between duplicates for all four samples.<sup>c</sup> Mean values after eliminating the data from Collaborator 1.**Table 6. Reproducibility of water determination within laboratories in mg per cigarette**

Coll. No.	Cambridge Filter			$\alpha$ -Cellulose Filter		
	Type A		All <sup>b</sup>	Type A		All
	Std Dev.	C. V. % <sup>a</sup>	Std Dev.	Std Dev.	C. V. %	Std Dev.
1	0.24	11.8	0.17	0.86	15.8	1.49
2	0.44	11.0	0.35	1.99	29.3	2.00
3	0.19	4.9	0.35	0.46	13.3	0.63
5	0.43	10.3	0.29	0.08	2.5	0.06
9	0.33	9.0	0.27	1.57	13.5	0.41
11	0.27	4.8	0.15	1.15	15.1	1.17
20	0.16	5.8	0.13	2.08	19.8	0.82
22	0.23	4.8	0.24	0.61	8.0	0.83
23	0.29	7.1	0.24	0.94	14.8	0.71
24	0.08	3.3	0.06	0.41	7.9	0.36
Mean	0.26	7.3	0.23	1.02	14.0	0.85

<sup>a</sup> C. V. % = Coefficient of variation in %.<sup>b</sup> All = Standard deviation calculated from the differences between duplicates for all four samples.

ber of puffs per sample and TPM, nicotine, and water per cigarette are shown in Tables 3, 4, 5, and 6, respectively. All analyses on Type A cigarettes were performed in quadruplicate so that the precision of measurements on one cigarette could be measured. The other three cigarettes were analyzed in duplicate. For the columns marked "All," the standard deviations were calculated from

the difference between duplicates for the four cigarettes, with the four analyses on Type A as two sets of duplicates.

Only in the water analysis were there any marked and consistent differences in precision between the two filter systems. Here the intralaboratory precision when the Cambridge filter was used was significantly better than that obtained with the

**Table 7. Within and between laboratory coefficients of variation in per cent for type A cigarettes**

	Cambridge Filter		$\alpha$ -Cellulose Filter	
	Av. within Labs.	Between Labs.	Av. within Labs.	Between Labs.
No. puffs	2.00	6.4	2.01	6.1
TPM	3.05	5.2	2.71	4.6
Nicotine	3.40	7.6	2.61	14.5
Water	7.3	31.9	14.0	37.4

$\alpha$ -cellulose filter in most of the laboratories. Within-laboratory and between-laboratory coefficients of variation for Type A cigarettes are tabulated in Table 7. Except for

TPM, the within-laboratory precision is markedly better than that between laboratories.

It is recommended<sup>1</sup> that more information be obtained from each collaborator as to the type and characteristics of his smoking machine and any other variables which might have affected the precision adversely, and that the present data then be re-evaluated to try to obtain some information on the causes of the variability and on what steps should be taken to obtain better precision between laboratories.

<sup>1</sup> This recommendation was approved by the General Referee and by Subcommittee A, and was accepted by the Association. See *This Journal*, 45, 120 (1962).